Bench-Scale Demonstration of Hot-Gas Desulfurization Technology

Quarterly Report

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U.S. Department of Energy
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Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

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By
Research Triangle Institute
P. O. Box 12194
Research Triangle Park, North Carolina 27709

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1.0 INTRODUCTION AND SUMMARY

The U.S. Department of Energy (DOE), Morgantown Energy Technology Center (METC), is sponsoring research in advanced methods for controlling contaminants in hot coal gasifier gas (coal gas) streams of integrated gasification combined-cycle (IGCC) power systems. The programs focus on hot-gas particulate removal and desulfurization technologies that match or nearly match the temperatures and pressures of the gasifier, cleanup system, and power generator. The work seeks to eliminate the need for expensive heat recovery equipment, reduce efficiency losses due to quenching, and minimize wastewater treatment costs.

Hot-gas desulfurization research has focused on regenerable mixed-metal oxide sorbents which can reduce the sulfur in coal gas to less than 20 ppmv and can be regenerated in a cyclic manner with air for multicycle operation. Zinc titanate (Zn₂TiO₄ or ZnTiO₃), formed by a solid-state reaction of zinc oxide (ZnO) and titanium dioxide (TiO₂), is currently one of the leading sorbents. Overall chemical reactions with Zn₂TiO₄ during the desulfurization (sulfidation)-regeneration cycle are shown below:

Sulfidation:
$$Zn_2TiO_4 + 2H_2S \rightarrow 2ZnS + TiO_2 + 2H_2O$$

Regeneration:
$$2ZnS + TiO_2 + 3O_2 \rightarrow Zn_2TiO_4 + 2SO_2$$

The sulfidation/regeneration cycle can be carried out in fixed-bed, moving-bed, or fluidized-bed reactor configuration, and all three types of reactors are slated for demonstration in the DOE Clean Coal Technology program. The fluidized-bed reactor configuration is most attractive because of several potential advantages including faster kinetics and the ability to handle the highly exothermic regeneration to produce a regeneration offgas containing a constant concentration of SO₂. However, a durable

attrition-resistant sorbent in the 100- to 400- μ m size range is needed for successful fluidized-bed operation.

The SO₂ in the regeneration offgas needs to be disposed of in an environmentally acceptable manner. Options for disposal include recycle to the gasifier in which an in-bed desulfurization sorbent such as dolomite or limestone is being employed, conversion to sulfuric acid, and conversion to elemental sulfur. All three options are being pursued and/or proposed in the Clean Coal Technology program. Elemental sulfur recovery is the most attractive option because sulfur can be easily transported, stored, or disposed. However, elemental sulfur recovery using conventional methods from an offgas containing low levels of SO₂ (typically 3%) is an expensive proposition. An efficient, cost-effective method is needed to convert the SO₂ in the regenerator offgas directly to elemental sulfur.

Research Triangle Institute (RTI) with DOE/METC sponsorship has been developing zinc titanate sorbent technology since 1986. In addition, RTI has been developing the Direct Sulfur Recovery Process (DSRP) with DOE/METC sponsorship since 1988. Fluidized-bed zinc titanate desulfurization coupled to the DSRP is currently the most advanced and attractive technology for sulfur removal/recovery for IGCC systems, and it has recently been proposed in a Clean Coal Technology project.

RTI has developed a durable fluidized-bed zinc titanate sorbent, ZT-4, which has shown excellent durability and reactivity over 100 cycles of testing at 750 to 780°C. In bench-scale development tests, it consistently reduced the H₂S in simulated coal gas to <20 ppmv and demonstrated attrition resistance comparable to fluid cracking catalysts. The sorbent is manufactured by a commercially scalable granulation technique using commercial equipment available in sizes up to 1,000 L. The raw materials used are relatively

inexpensive, averaging about \$1.00/lb. It is anticipated that the impact on cost of electricity (COE) due to sorbent replacement for attrition will be less than 0.5 mil/kWh. ZT-4 has recently been tested independently by the Institute of Gas Technology (IGT) for Enviropower/Tampella Power, and showed no reduction in reactivity and capacity after 10 cycles of testing at 650°C.

In the DSRP SO₂ is catalytically reduced to elemental sulfur using a small slip stream of the coal gas at the pressure and temperature conditions of the regenerator offgas. A near-stoichiometric mixture of offgas and raw coal gas (2 to 1 mol ratio of reducing gas to SO₂) reacts in the presence of a selective catalyst to produce elemental sulfur directly:

$$2H_2 + SO_2 \rightarrow (1/n)S_n + 2H_2O$$

 $2CO + SO_2 \rightarrow (1/n)S_n + 2CO_2$
 $CO + H_2O \rightarrow CO_2 + H_2$

The above reactions occur in Stage I of the process, and convert up to 96% of the inlet SO_2 to elemental sulfur, which is recovered by cooling the outlet gas to condense out the sulfur. Adjusting the stoichiometric ratio of coal gas to regenerator offgas to 2 at the inlet of the first reactor also controls the Stage I effluent stoichiometry since any H_2S and COS produced (by the reactions: $3H_2 + SO_2 \rightarrow H_2S + 2H_2O$, and $3CO + SO_2 \rightarrow COS + 2CO_2$) yields an $(H_2S + COS)$ to SO_2 ratio of 2 to 1. The effluent stoichiometry plays an important role in the Stage II DSRP reactor (operated at 275 to 300°C), where 80% to 90% of the remaining sulfur species is converted to elemental sulfur most probably via $COS + H_2O \rightarrow H_2S + CO_2$ and $2H_2S + SO_2 \rightarrow (3/n)S_n + 2H_2O$. The overall sulfur recovery is projected at 99.5%.

The DSRP technology is also currently at the bench-scale development stage with a skid-mounted system ready for field testing. Very recently, the process has been extended to fluidized-bed operation in the Stage I reactor. Fluidized-bed operation has proved to be very successful with conversions up to 94% at space velocities ranging from 8,000 to 15,000 scc/cc·h. Overall conversion in the two stages following interstage sulfur and water removal has ranged up to 99%.

A preliminary economic study for a 100 MW plant in which the two-stage DSRP was compared to conventional processes indicated the economic attractiveness of the DSRP. For 1% to 3% sulfur coals the installation costs ranged from 25 to 40 \$/kW and the operating costs ranged from 1.5 to 2.7 mil/kWh.

Through bench-scale development, both fluidized-bed zinc titanate and Direct Sulfur Recovery Process (DSRP) technologies have been shown to be technically and economically attractive. The demonstrations to date, however, have only been conducted using simulated (rather than real) coal gas and simulated regeneration off-gas. Thus, the effect of trace contaminants in real coal gases on the sorbent and DSRP catalyst is currently unknown. Furthermore, the zinc titanate work to date has emphasized sorbent durability development rather than database development to permit design of large-scale reactors. Discussions with fluidized-bed experts have indicated that data from a larger reactor than the present are required for scaleup, especially if the material does not have particle sizes similar to fluid catalytic cracking catalysts (typically ~80 μm). The fluidized-bed zinc titanate technology uses 100- to 400-μm particles. Finally, the zinc titanate desulfurization unit and DSRP have not been demonstrated in an integrated manner.

The goal of this project is to continue further development of the zinc titanate desulfurization and DSRP technologies by

- Scaling up the zinc titanate reactor system;
- Developing an integrated skid-mounted zinc titanate desulfurization-DSRP reactor system;
- Testing the integrated system over an extended period with real coal-gas from an operating gasifier to quantify the degradative effect, if any, of the trace contaminants present in coal gas;
 - Developing an engineering database suitable for system scaleup; and
- Designing, fabricating and commissioning a larger DSRP reactor system capable of operating on a six-fold greater volume of gas than the DSRP reactor used in the bench-scale field test.

2.0 TECHNICAL DISCUSSION

2.1 FIELD TESTING OF ZTFBD/DSRP AT METC

2.1.1 SUMMARY OF OCTOBER FIELD TEST

This section summarizes the highlights and accomplishments of the October slipstream test run of the Zinc Titanate Fluid Bed Desulfurization/Direct Sulfur Recovery Process (ZTFBD/DSRP) Mobile Laboratory at the Department of Energy's Morgantown Energy Technology Center (METC). The excellent cooperation and assistance by METC personnel to RTI field staff contributed significantly to the success of the test. Although the run had to be shortened due to mechanical problems with METC's gasifier, there was sufficient on-stream time to demonstrate highly successful operation of both the zinc titanate fluid bed desulfurization and the DSRP with actual coal gas. Also, the multimetals, NH₃, and HCI/HF impinger trains were successfully used during the run to determine the level of trace contaminants. No significant effect of the contaminants was detected on either the ZTFBD or DSRP over the 70 hours of the run.

The process equipment in the ZTFBD unit worked extremely smoothly in both the sulfidation and regeneration modes. The fluidizable zinc titanate formulation ZT-4L demonstrated 99+ percent removal of hydrogen sulfide (H₂S) from actual coal gas over 3 cycles, and up to 20 lbs sulfur per 100 lbs sorbent loading capacity. It also demonstrated consistent, smooth regeneration behavior. The DSRP unit for the most part also ran smoothly with actual coal gas. As planned, to obtain extended operation of the DSRP, provisions were made to produce simulated regeneration off-gas using liquid sulfur dioxide (LSO₂). This equipment worked very well. During periods of lined-out operation, the DSRP had concentrations of sulfur compounds in the exit gas corresponding to up to 99%+

conversion in Stage 1 and 95-96 percent overall conversion for the two-stage system. This suggested problems of reverse Claus in Stage 2. For a commercial system one stage may be sufficient thus further improving the already attractive economics of DSRP.

The DSRP unit was also successfully run in a fully-integrated mode using actual regeneration off-gas. However, the ZTFBD run times in this mode were fairly short (due to a limited capacity to produce actual off-gas) and because of the longer time constants of the DSRP unit, lined-out operation could not be achieved.

The DSRP unit did experience some plugging problems that resulted in unscheduled outages. The very small flow of coal gas used by the DSRP means that the high temperature, high pressure control valve that is required is very small. It tended to plug if there was any particulate matter in the coal gas. The high temperature ceramic filter on the RTI equipment was apparently not completely effective and suggests that for small equipment a stainless steel filter may be better. There were also some problems with sulfur plugging in the cold end of the unit that required depressurization and disassembly to unblock. This suggests that maintaining the exit gases hot will be necessary for continuous operation, free of plugs.

There was one time period when it appeared that the "RTI Interface Line" that METC had installed between the Modular Gas Cleanup Rig (MGCR) and the RTI trailer had become plugged. Efforts to clear the line while maintaining system pressures were not effective. Subsequently, the line was taken out of service and some particulates were removed from the line when it was depressurized and disassembled. Since this cleanup did not reveal any large amount of solids it remains unclear as to why there was apparently no flow for awhile.

By RTI's calculations, summarized below, there was coal gas available at the RTI trailer for a total of 70 hours during the shortened October run. RTI was taking coal gas for 45.5 of those hours, for a utilization factor of 65 percent.

The following are highlights of the day-to-day operation of the bench-scale process equipment.

Monday, October 24, 1994

- 00:01 Startup sequence initiated by METC.
- 08:45 RTI personnel started heating up furnaces, etc. in RTI trailer.
- 16:00 The operators for the Modular Gas Cleanup Rig (MGCR) started hot nitrogen flowing through their unit, and also into the RTI Interface line connected to the RTI trailer.
 Hot nitrogen began flowing into RTI's ZTFBD unit.
- 16:40 Coal gas started flowing into MGCR and simultaneously into the interface line; start of Sulfidation Run #1.
- 22:30 H₂S breakthrough at 1100 ppm (according to the Carle GC on sample point A-2).

 Stopped Sulfidation Run #1 (total time was 5:50).
- 23:14 Started Regeneration Run #1 with integrated operation of the DSRP.

Tuesday, October 25, 1994

- 00:32 End of Regeneration Run #1 (total time 1:18).
- 02:05 Began Sulfidation Run #2.
- 09:08 H₂S breakthrough at approximately 1000 ppm. End Sulfidation Run #2 (total time was 7:03).
 - Set up trace contaminant sampling trains.
- 12:20 Started coal gas flowing for Regeneration Run #2.

- 12:30 Started air flowing for Regeneration Run #2. The acid gas trace contaminant sampling train was used on Stage 1 off-gas (TCT-5) and the multi-metals sampling train was used on the regeneration off-gas (TCT-3)
- 13:46 End of Regeneration Run #2 (total time 1:16).

At this point, the DSRP was depressurized and hardware modifications were made to move the sample point for the Western SO₂ analyzer from A-4 to A-5A (which is more suitable for when DSRP is run using liquid sulfur dioxide [LSO₂]). In order that both sample points would be available in the future without depressurization again, sample conditioning equipment was cannibalized from sample point A-5 for use with A-5A.

During this time period it was also determined that the Omega mass flow controller (being used as a flow meter) installed on the DSRP (designated FT-240 on the P&ID) was no longer functioning properly and it was removed from the system.

- 18:13 Started LSO₂ flowing and adjusted controller by comparing with Western SO₂ analyzer.
- 18:43 Started coal gas flowing into DSRP for start of DSRP/LSO₂ Run #1.
- 23:30 Problems with maintaining coal gas flow were noted; assumed problem was plugging of Badger control valve FCV-2.

Wednesday, October 26, 1994

- 00:12 Stopped coal gas flow; took nitrogen through interface line. End of DSRP/LSO₂ Run #1 (total time 6 hrs).
- 01:15 Started Sulfidation Run #3.

10:10 Continuing Sulfidation Run #3; some irregularity noted in automatic control of Badger valve FCV-1. Coincident with plugging problems noted by MGCR operators. Nitrogen was substituted for coal gas in MGCR (and hence, in interface line, as well) for approximately 1 hour.

During this time the multi-metals trace contaminant sampling train was running on raw coal gas (TCT-1), and the acid gas train was running on the stage one off-gas (TCT-5).

- 11:10 MGCR advised RTI that coal gas was flowing in MGCR again, and that gasifier had started taking chloride-doped coal.
- 11:30 Coal gas flow through the interface line was interrupted at RTI request so that maintenance could be performed on FCV-2 (DSRP).
- 12:11 Coal gas flow to sulfidation run restarted.
- 14:10 End of Sulfidation Run #3 with H₂S breakthrough of greater than 3000 ppm H₂S (total time: 13 hrs).

Set up trace contaminant sampling trains.

16:10 Coal gas flow to DSRP started for DSRP/LSO₂ Run #2.

The ammonia trace contaminant sampling train was run on stage one off-gas (TCT-5), and the acid gas train was run on stage two off-gas (TCT-6).

17:49 Run was stopped when it was determined that little or no coal gas was actually flowing. Various means were attempted to clear the line; evidence suggested that METC Interface line (or orifice) was plugged. DSRP was left hot with 67 SLPM of nitrogen flowing through it.

Thursday, October 27, 1994

- A.M. METC and EG&G staff worked to clear the Interface Line. RTI worked to re-install sample conditioning equipment at Sample Point A-5 (cannibalized from Point A-3) in order to run Carle GC on mixture of regeneration gas and coal gas as an additional aid to determination of coal gas flow rate.
- 12:00 Coal gas was available again, through the Interface Line. RTI could not operate DSRP, however, due to sulfur plugging in cold end. Attempted to clear the plug; eventually switched to alternative flow path in cold end train #2. Drained sulfur condenser #1. No sulfur was collected in condensers #2 and #3.
- 16:26 Started liquid SO₂ flowing for DSRP/LSO₂ Run 3.
- 16:27 Started coal gas flowing. The multi-metals trace contaminant sampling train was run on the first stage off-gas (TCT-5).
- 20:10 Plugging in DSRP noted; stopped DSRP/LSO₂ Run 3. Determined that cold end train #2 had plugged.

Worked to clear plugging from cold end.

23:19 Started liquid SO₂ and coal gas flowing for DSRP/LSO₂ Run 4.

Friday, October 28, 1994

- 05:12 Safety relief valve on Sulfur Condenser #1 popped. Coal gas released to equipment room; detected by toxic gas monitoring system. Coal gas flow stopped; end of DSRP/LSO₂ Run 4.
- 10:00 MGCR advised that they were going off coal gas for approximately 24 hours. Also, gasifier operators decided to stop feeding coal and go into a "hot hold" with no coal gas production for approximately 24 hours.

RTI determined that the #1 sulfur condenser was leaking process gas into the water jacket. Condenser was drained of sulfur, removed from service and disassembled. The leak was determined to be in the coil; a new coil was fabricated and the condenser was reassembled and reinstalled by 18:00. Sulfur was also drained from condenser #3; none was collected in condenser #2.

21:00 METC advised RTI that the gasifier team had decided to stop the run due to the discovery of a heat-deformed pipe in the ash-removal system.

RTI turned off heat to furnaces and heat tracing. Left a small nitrogen purge through the equipment. Bled off the unused liquid SO₂ to the incinerator stack.

Saturday, October 29, 1994

All Day METC operating crews had finished up during the night. RTI staff cleaned up and put away equipment. Finished purging SO₂. Diluted reagents from the trace sampling project were disposed of. Sorbent, catalyst, and recovered sulfur samples were removed and packed for transport back to North Carolina. Some instruments and tools were removed and packed for transport. Cooling water was removed from the system. All equipment was depressurized; cylinders were capped, power to the control panel and the equipment skids was shut off. The HVAC units were left on with set points of 55 °F and 80 °F for heating and cooling, respectively.

SUMMARY OF TOTAL HOURS:

Coal gas availability:

Monday 7:20

Tuesday 24:00

Wednesday 17:00

Thursday 12:00

Friday 10:00

TOTAL 70 hrs

RTI on-stream operation (taking coal gas):

Sulf. #1 5:50

Regen. #1 1:15

Sulf. #2 7:00

Regen. #2 1:15

DSRP/LSO₂ #1 6:00

Sulf. #3 7:00

DSRP/LSO₂ #2 1:30

DSRP/LSO₂ #3 3:40

DSRP/LSO₂ #4 6:00

TOTAL 45.5 hrs

Highlights of results of the tests are described below.

2.1.2 RESULTS

During the October test, 3 sulfidation cycles (~25 hours), 2 integrated DSRP tests (2.5 hours) and 4 simulated DSRP tests (18 hours) were conducted. Test conditions and main results of the testing are presented. A typical METC coal gas composition is shown in Table 1.

Table 1. METC Gasifier Coal Gas Composition (volume%)

CH₄
$H_2 \dots 14.9$
CO ₂ 11.5
CO 9.87
H ₂ O 11.0
H ₂ S 0.1 - 0.75
N ₂ Balance
HCl 5 - 80 ppmv
As <10 μg/m ³
Se 16 μg/m³
Hg $<2 \mu g/m^3$
NH ₃ ~ 800 ppmv

Points to note in Table 1 are that H₂S and HCI are quite variable because of coal's variability and at times doping of the coal with salt to increase the HCI level for a Shell slipstream test on chloride removal. The ammonia analysis is an estimated value because of coal gas line plugging during the sampling. The trace contaminant values were below or near the detection limit of our sampling system.

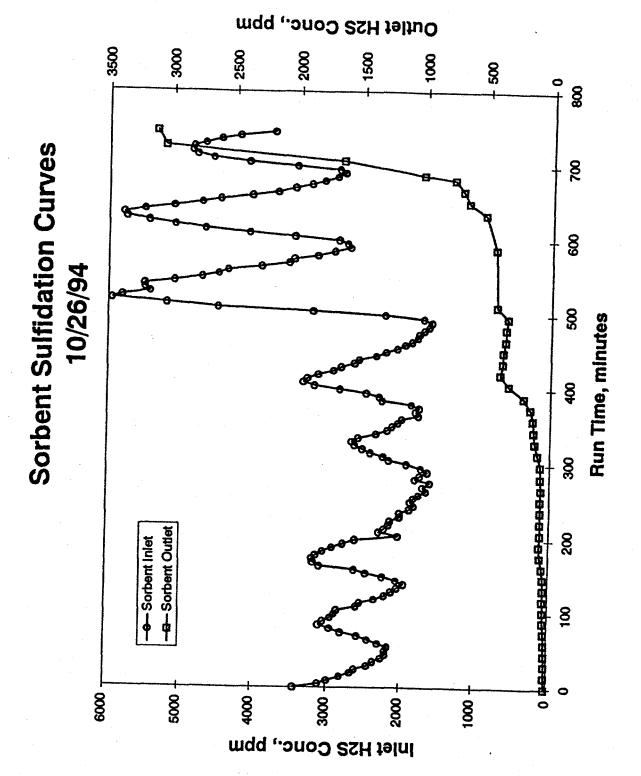
Test conditions for sulfidation and regeneration of ZT-4L over the 2.5 cycles are shown in Table 2. Under these conditions, an H_2S breakthrough curve during cycle 3 is shown in Figure 1. The sorbent exhibited excellent removal efficiency and capacity even with highly variable inlet H_2S values. Sorbent regeneration, which went very smoothly, is shown in Figure 2. Note that with 2.25% O_2 in the inlet, nearly exactly 2/3, i.e. ~1.5% SO_2

Table 2. ZT-4 Reactor Conditions

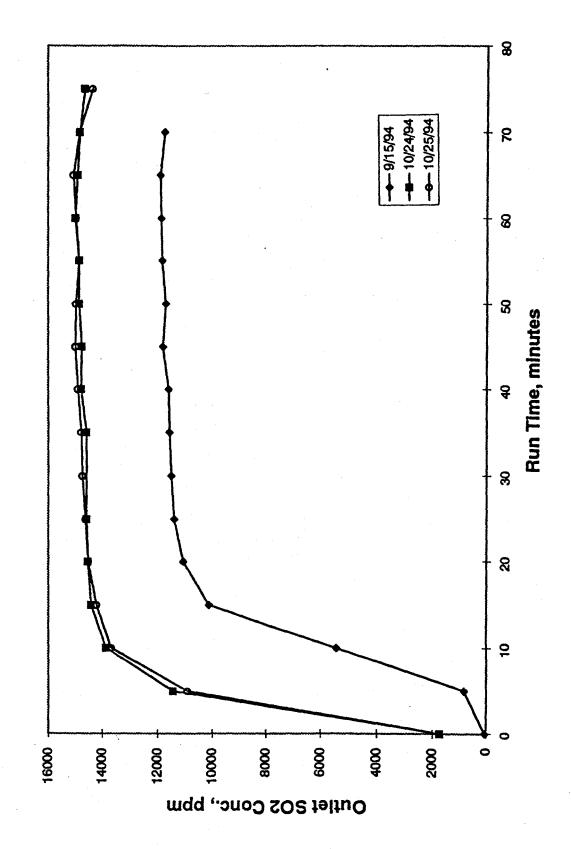
(3.0-in. Reactor; 600 g Sorbent loaded)

	Sulfidation	Regeneration
Temperature (°C)	009	730
Pressure (psig)	260	260
U _s (cm/s)	4.3	4.9
Gas	Coal Gas	2.25% O ₂ in N ₃

Figure 1



Sorbent Regeneration Curves



is obtained in the off-gas during the October run indicating essentially no sulfation. The properties of the fresh and 3-cycle (sulfided) materials are compared in Table 3. The surface area of the sulfided material is higher and the pore volume is lower. The material significantly improved in attrition resistance over the 3 cycles. The TGA capacity did not change from fresh to used material and was at 20 lb/100 lb sorbent. The trace contaminants were below the detection limit, although chloride up to 38 ppmv possibly some selenium up to 15 ppmv do appear to accumulate on the sorbent. Preliminary indications are that the actual coal gas - sulfided sorbent required somewhat higher temperature for regeneration then a sorbent sulfided with simulated coal gas. This may be due to reactions of one or more of the contaminants in coal gas with the sorbent. This problem needs further investigation.

The test conditions for the DSRP Stage 1 reactor are shown in Table 4. As indicated earlier, two integrated ZTFBD-DSRP and four simulated SO₂ - DSRP tests were conducted using the METC gasifier coal gas. During the integrated tests, it was not possible to obtain lined out operation in the short duration due to a larger time constant (~2 hours) of the DSRP. However, three of the simulated SO₂ - DSRP tests were highly successful. One of the simulated SO₂ - DSRP test was not successful due to plugging of the coal gas line from METC's gasifier. The results of the successful DSRP tests are shown in Table 5. Note that in Run 1 with fresh catalyst extremely high sulfur conversion (up to 99.7%) is achieved. Some selectivity towards H₂S is seen in later runs, although even in these tests a 96% sulfur conversion level is achieved. This is an excellent performance by DSRP.

Table 3. Properties of Fresh and Reacted ZT-4L

	Fresh	3-Cycle Sulfidated
Expsoure time (h)	0	Coal Gas (25); Temp. (>100)
Surface area (m²/g)	3.2	7.56
Pore volume (cm³/g)	0.234	0.11
Pore diameter (Å)	2,500	1,800
Particle size (μm)	112	95
Air-jet attrition (%) 5-h loss 20-h loss	16 36	1.4 6.2
Compacted density (lb/ft³)	91.6	107
Zn/Ti	1.35 ± 0.05	1.42 ± 0.05
TGA capacity (g/100 g)	21.0	20.5
As (µg/g)	NN	<10
Se (µg/g)	NM	15
Cl (µg/g)	NM	38

NM = not measured.

Table 4. DSRP Stage I Reactor Conditions

Temperature (°C)	550 - 610
Pressure (psig)	260
Space velocity (scc/cc•h)	4,560
Reactor diameter (in.)	3.0
Inlet SO ₂ (%)	1.8

Table 5. Stage 1 DSRP Results During Lined-Out Operation with Simulated SO₂.

Aun No. 1 99.4 0.0 99.4 36 99.4 0.0 99.4 40 99.5 0.0 99.5 44 99.5 0.0 99.5 48 99.5 0.0 98.5 56 98.0 0.0 98.5 56 98.0 0.0 98.5 117 99.6 1.2 98.4 121 100.0 4.1 95.9 124 100.0 2.4 97.6 Aun No. 4 100.0 4.9 95.1 179 100.0 4.9 95.1 183 100.0 4.9 95.1 183 100.0 4.9 95.1 183 100.0 4.9 95.1 183 100.0 4.9 95.1 199 100.0 4.7 95.3	Run Time (min)	SO ₂ Conversion (%)	SO ₂ Conversion to H ₂ S (%)	SO ₂ Conversion to Sulfur (%)
32 99.4 0.0 36 99.4 0.0 40 99.5 0.0 44 99.7 0.0 48 99.5 0.0 52 98.5 0.0 56 98.0 0.0 117 99.6 1.2 121 100.0 4.1 125 100.0 4.9 175 100.0 4.9 179 100.0 4.9 183 100.0 4.7 199 4.7	Run No. 1			
36 99.4 0.0 40 99.5 0.0 44 99.7 0.0 48 99.5 0.0 52 98.5 0.0 56 98.0 0.0 117 99.6 1.2 121 100.0 4.1 125 100.0 4.9 175 100.0 4.9 179 100.0 4.1 183 100.0 4.7 199 100.0 4.7	32	99.4	0.0	99.4
40 99.5 0.0 44 99.7 0.0 48 99.5 0.0 52 98.5 0.0 56 98.0 0.0 117 99.6 1.2 121 100.0 4.1 125 100.0 4.9 175 100.0 4.9 179 100.0 4.1 183 100.0 4.7 189 100.0 4.7	36	99.4	0.0	99.4
44 99.7 0.0 48 99.5 0.0 52 98.5 0.0 56 98.0 0.0 117 99.6 1.2 121 100.0 4.1 125 100.0 2.4 175 100.0 4.9 179 100.0 4.9 183 100.0 4.7 189 100.0 4.7	40	99.5	0.0	99.5
48 99.5 0.0 52 98.5 0.0 56 98.0 0.0 117 99.6 1.2 121 100.0 4.1 125 100.0 4.9 129 100.0 2.4 175 100.0 4.9 178 100.0 4.1 183 100.0 4.7 189 100.0 4.7	44	2.66	0.0	29.7
52 98.5 0.0 56 98.0 0.0 117 99.6 1.2 121 100.0 4.1 125 100.0 2.4 129 100.0 4.9 175 100.0 4.9 183 100.0 4.1 183 100.0 4.7 199 100.0 4.7	48	99.5	0.0	99.5
56 98.0 0.0 117 99.6 1.2 121 100.0 4.1 125 100.0 4.9 129 100.0 2.4 175 100.0 4.9 179 100.0 4.1 183 100.0 4.7 199 100.0 4.7	52	98.5	0.0	98.5
11799.61.2121100.04.1125100.04.9129100.02.4175100.04.9178100.04.9183100.04.9199100.04.7	56	98.0	0.0	98.0
11799.61.2121100.04.1125100.02.4129100.04.9175100.04.1183100.04.9183100.04.9199100.04.7	Run No. 3			
121100.04.1125100.04.9129100.02.4175100.04.9183100.04.9199100.04.7	117	9.66	1.2	98.4
125100.04.9129100.02.4175100.04.9183100.04.1199100.04.7	121	100.0	4.1	95.9
129 100.0 2.4 175 100.0 4.9 179 4.1 183 100.0 4.9 199 100.0 4.7	125	100.0	4.9	95.1
175 100.0 4.9 179 4.1 183 100.0 4.9 199 4.7	129	100.0	2.4	97.6
100.0 4.9 100.0 4.1 100.0 4.9 100.0 4.7	Run No. 4			
100.0 100.0 4.9 100.0	175	100.0	4.9	95.1
100.0 4.9 100.0 4.7	179	100.0	4.1	95.9
100.0	183	100.0	4.9	95.1
	199	100.0	4.7	95.3

The fresh and used catalyst properties are shown in Table 6. The used catalyst showed better crush strength but a lower surface area. Also, chloride is picked up by the catalyst - but does not appear to affect its activity significantly even at 300 ppmv level. No other trace contaminants are detected in the catalyst.

To conclude, both ZT-4L and DSRP showed very promising results in short-term testing with actual coal gas. The test of 160 hours needs to be completed to evaluate longer term effects.

2.1.3 SUGGESTED MODIFICATIONS TO RTI TRAILER FOR POTENTIAL FUTURE SLIPSTREAM TESTS

Based on the interrupted October field test, the following modifications were suggested to METC to complete the test.

- Convert DSRP system to single stage and run continuously on simulated SO₂
 with actual coal gas.
- Temperature/pressure corrected orifice flow meter for coal gas.
- Process control computer to input SO₂ mass flow and hydrogen and CO concentrations and output required coal gas flow set point to the coal gas flow control valve.
- Modify sulfur condenser to have a separate pot with a heating jacket and nonfreezing drain valve.
- Install Pall metal HTHP filter on coal gas line to trailer.
- Install HTHP back-pressure control valve system.
- Install heating system for DSRP off-gas vent.
- Install dedicated DDAS terminal to obtain H₂, CO signals from METC.

Table 6. DSRP Stage I Catalyst

	Fresh	Used
Size (in.)	1/8	1/8
Crush strength (lbf/mm)	2.0	2.5
Surface area (m²/g)	208	158
Exposure (hour)	0	Coal Gas (20); Temp. (>100)
CI (ppmw)	21	300
As (ppmw)	<11	<10
Se (ppmw)	<10	<10

Install large knockout pot for DSRP off-gas vent.

2.2 SCALED-UP DSRP REACTOR SYSTEM

A meeting was held with Enviropower and based on the METC field test results it was decided to develop a single-stage fixed-bed DSRP reactor system rather than a two-stage system. Significant accomplishments were made during the quarter towards the goal of supplying the reactor system to Enviropower. These are highlighted below:

- Preliminary schedule for system delivery was prepared.
- A meeting was planned and scheduled at the Enviropower pilot plant on January 9 and 10, 1995.
- Prior to Christmas, the following documents were delivered to Enviropower in preparation of a detailed face to face meeting:
 - draft process flow diagram (PFD);
 - process material balance;
 - draft process and instrumentation diagrams (PID).
- Preliminary mechanical designs for reactors and other vessels and preliminary electrical diagrams were prepared.
- Vendors for long lead items were contacted and preliminary quotes were obtained.

3.0 PLANS FOR NEXT QUARTER

- 1. Detailed design of scaled-up DSRP System based on meeting with Enviropower.
- 2. Second design meeting with Enviropower.
- 3. Preparation for resumption of field-test at METC in late spring and summer.
- Critical path management (CPM) charts for field test at METC and for scaledup DSRP System.
- Implementation of CPM with ordering of all equipment requiring long delivery time.
- 6. Preparation for Houston AlChE paper.

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